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## (54) RARE-EARTH RESIN MAGNET HAVING LOW COERCIVE FORCE

### (57)Abstract:

PURPOSE: To obtain a rare-earth resin magnet which has a low coercive force and can be excellently magnetized in multiple poles without deteriorating its magnetic characteristic as much as possible by mixing quenched powder having a chemical composition close to the stoichiometric composition Nd<sub>2</sub>Fe<sub>14</sub>B and the powder for switching spring magnets and forming the magnet by binding the mixture with a resin.

CONSTITUTION: Quenched powder containing  $\pm 0.5$ at.% Nd<sub>12</sub> which is close to the stoichiometric composition Nd<sub>2</sub>Fe<sub>14</sub>B (Nd<sub>12</sub>Fe<sub>82</sub>F<sub>6</sub>) and having a coercive force Hc<sub>J</sub>&gt;10kOe is mixed with the powder for switching spring magnets in which the size of crystal grains is controlled to 20-50mm and which is composed of a soft magnetic phase and hard magnetic phase. Then a rare-earth resin magnet is obtained by binding the mixture with a resin. The magnet thus obtained has a coercive force of  $\geq 320$ kA/m (4kOe) and <795kA/m (10kOe). In addition, the permeance factor P<sub>c</sub> of the magnet is controlled to  $\geq 5$ . Therefore, a rare-earth resin magnet which has a high matching property with the magnetic characteristics, such as the (BH)<sub>max</sub>, Br,  $\Delta$ Br/ $\Delta$ T(%/K),  $\Delta$ Hc<sub>J</sub>/ $\Delta$ T (%/K), etc., even in a low coercive force region and can be excellently magnetized in multiple poles can be obtained easily.

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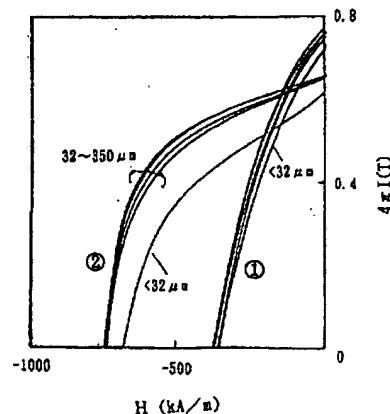
(54)【発明の名称】 低保磁力希土類樹脂磁石

(57)【要約】

【目的】 低保磁力領域であっても ( $BH$ )<sub>max</sub>,  $B_r$ ,  $\Delta B_r / \Delta T$  (%/K),  $\Delta H_{c1} / \Delta T$  (%/K) など磁気特性の整合性の高い多極着磁性に優れた希土類樹脂磁石を提供する。

【構成】 Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成 (Nd<sub>1</sub>, Fe<sub>13</sub>, B<sub>6</sub>) 付近の Nd<sub>1</sub>, ± 0.5at%、保磁力  $H_{c1}$  ≈ 10kOe の急冷粉体と結晶粒が 20~50nm に制御されたソフト磁性相とハード磁性相から構成される交換スプリング磁石粉体とを混合し、これを樹脂で固めて希土類樹脂磁石とする。

(低 Nd量と化学量論 Nd量急冷磁石の  
薄帯粒子径による減磁曲線の変化)



① Nd<sub>3.5</sub>Dy<sub>1</sub>Fe<sub>13</sub>C<sub>0.3</sub>Ga<sub>1</sub>B<sub>18.5</sub>

② Nd<sub>12.3</sub>Fe<sub>76.5</sub>C<sub>0.5</sub>B<sub>5.7</sub>

## 【特許請求の範囲】

【請求項1】Nd<sub>x</sub>Fe<sub>y</sub>B化学量論組成(Nd<sub>x</sub>Fe<sub>y</sub>,B<sub>z</sub>)付近のNd<sub>x</sub>±0.5at%、保磁力H<sub>c1</sub>≈10kOeの急冷粉体と、結晶粒が20~50nmに制御されたソフト磁性相とハード磁性相から構成される交換スプリング磁石粉体とを混合し、これを樹脂で固めた320kA/m(4kOe)以上、795kA/m(10kOe)未満の低保磁力希土類樹脂磁石。

【請求項2】結晶粒が32μm以下を含むソフト磁性相とハード磁性相から構成される交換スプリング磁石粉体を、前記交換スプリング磁石粉体よりも粉体粒子径が大きなNd<sub>x</sub>Fe<sub>y</sub>,B化学量論組成(Nd<sub>x</sub>Fe<sub>y</sub>,B<sub>z</sub>)付近の急冷粉体と混合し、これを樹脂でリング状に固めた320kA/m(4kOe)以上、795kA/m(10kOe)未満の低保磁力希土類樹脂磁石。

【請求項3】バーミアンス係数Pcを5以上とした請求項1または請求項2記載の低保磁力希土類樹脂磁石。

## 【発明の詳細な説明】

## 【0001】

【産業上の利用分野】本発明は、Nd<sub>x</sub>Fe<sub>y</sub>,B化学量論組成(Nd<sub>x</sub>Fe<sub>y</sub>,B<sub>z</sub>)付近のNd<sub>x</sub>±0.5at%、保磁力H<sub>c1</sub>≈10kOeの急冷粉体と結晶粒が20~50nmに制御されたソフト磁性相とハード磁性相から構成される交換スプリング磁石粉体とを混合し樹脂で固める低保磁力希土類樹脂磁石に関する。

## 【0002】

【従来の技術】2:14:1に近い割合のNd<sub>x</sub>Fe<sub>y</sub>,B三元系溶湯合金をメルトスパンした急冷粉体は結晶化温度約870KのNd<sub>x</sub>Fe<sub>y</sub>,B化合物を主相とする準安定状態の材料で、急冷の程度に応じOVER-QUENCH, OPTIMUM-QUENCH, UNDER-QUENCH状態に区分され、OPTIMUM-QUENCH状態で高保磁力(H<sub>c1</sub>)となる。

【0003】J.F.HerbestらはRare earth-Iron-Boron Materials: A New Era in Permanent Magnets, Ann. Rev. Mater. Sci., vol.16 p467(1986)で述べているようにNd<sub>x</sub>Fe<sub>y</sub>,B相を単磁区臨界寸法~300nm以下に結晶制御したOPTIMUM-QUENCH状態の急冷薄帯の残留磁化J<sub>r</sub>は0.8T、最大エネルギー積([BH]max) 111.5kJ/m<sup>3</sup>(14MGOe)としている。

【0004】上記、OPTIMUM-QUENCH状態の急冷薄帯を平均粒子径約150μm程度に粉碎した急冷粉体は、もっぱら真密度の80%程度まで一般にはリング形状に圧縮して樹脂で固定し、(BH)max~71kJ/m<sup>3</sup>(~9MGOe)の希土類樹脂磁石とする。この希土類樹脂磁石は1987年以来急速に実用化され、SmCo系に続く希土類樹脂磁石としての地位を得た。

【0005】一方、急冷粉体の実用化の進展とともに、Nd<sub>x</sub>Fe<sub>y</sub>,B化学量論組成(Nd<sub>x</sub>Fe<sub>y</sub>,B<sub>z</sub>)よりも低Nd量の急冷薄帯の研究も行われた。

## 【0006】HirosawaらはHigh Coercivity Iron-Rich

Rare earth Permanent Magnet Material Based on (Fe, Co), B-Nd-M(M=A1, Si, Cu, Ga, Ag, Au). 37th MMM(1992) FC-10で、Nd<sub>x</sub>Fe<sub>y</sub>,B化学量論組成より低Nd量における系にGaなどを1at%添加し結晶粒を微細制御することでOPTIMUM-QUENCH状態の急冷粉体とすればH<sub>c1</sub>が320kA/m(4kOe)程度が得られるとしている。

【0007】Nd量約4at%という低Nd量急冷粉体は磁気的に等方性で残留磁化J<sub>r</sub>と飽和磁化J<sub>s</sub>の比が0.50程度でなく、たとえばJ<sub>r</sub>/J<sub>s</sub>=0.75と高くなっている点が注目される。

【0008】上記急冷粉体の主相は微細結晶粒(30nm)である磁気的にソフトなFe<sub>y</sub>B準安定相がNd<sub>x</sub>Fe<sub>y</sub>,B相と結晶粒界を介した交換相互作用で繋がることでH<sub>c1</sub>がるとされ、Knellerらは、The Exchange-Spring Magnet: A New Material Principle for Permanent Magnets, IEEE Trans. Magn., 27 p3588(1991)にあるように、これに“Exchange-spring-magnet”交換スプリング磁石と名づけた。名の由来はソフト相Fe<sub>y</sub>Bとハード相Nd<sub>x</sub>Fe<sub>y</sub>,Bが交換相互作用で繋がり、ハード相が磁化反転しない限り逆磁界を取り除いてもソフト相の磁化がバネ(spring)のように可逆的に戻って元の磁化が得られる点にある。ふつう、このようにハード相とソフト相が存在すると磁化曲線には段が生じて磁気特性の低下を招く。しかし2相間に交換相互作用が働く程に結晶粒を微細化(20~50nm)すれば段は生じずに等方性であるにもかかわらず高J<sub>r</sub>が得られることが明らかになっている。

## 【0009】

【発明が解決しようとする課題】図8は合金組成Nd<sub>x</sub>(Fe<sub>y</sub>,Co<sub>z</sub>)balB<sub>6</sub>OPTIMUM-QUENCH急冷粉体を樹脂で固めた希土類樹脂磁石のNd量に対する磁気特性を示す。ただし磁界(Hm)4770kA/mパルス着磁後の磁気特性値である。またH<sub>k</sub>は磁化がB<sub>r</sub>の90%に達したときの減磁界で、H<sub>k</sub>/H<sub>c1</sub>は減磁曲線の角型性を表している。

【0010】Nd<sub>x</sub>Fe<sub>y</sub>,B化学量論組成におけるNd量は12at%であるが、(BH)max, H<sub>k</sub>/H<sub>c1</sub>, B<sub>r</sub>などの磁気特性はNd<sub>x</sub>Fe<sub>y</sub>,B化学量論組成付近の12~12.5at%で最も整合性が高い。Nd量が12.5at%を越えると高H<sub>c1</sub>となるが、(BH)max, H<sub>k</sub>/H<sub>c1</sub>, B<sub>r</sub>が低下する。

【0011】図9は上記希土類樹脂磁石の磁界(Hm)1908kA/m, 4770kA/mパルス着磁後の(BH)max, H<sub>k</sub>/H<sub>c1</sub>, B<sub>r</sub>などの磁気特性の比をH<sub>c1</sub>との関係で示す。

【0012】H<sub>c1</sub>が795kA/m(10kOe)を越えると磁石の着磁性が低下する。したがって、多極着磁することの多い希土類樹脂磁石はH<sub>c1</sub>, 795kA/m(10kOe)程度の化学量論組成に近いものが一般に使用される。

【0013】しかし、極間距離がさらに狭まると、十分な着磁磁界を発生させることが困難となりH<sub>c1</sub>, 795kA/m

(10kOe) でも着磁が困難になる。また、図3のように Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成よりも低 Nd量にすると αFe の析出をおさえ、組織の均質化を図るのが困難となるため H<sub>c1</sub>, (BH) max, Hk/H<sub>c1</sub>, Brなどの磁気特性が低下する。

【0014】以上のように、(BH) max, Br, ΔBr/ΔT (%/K), ΔH<sub>c1</sub>/ΔT (%/K)などの磁気特性をできる限り低下させずに、多極着磁性に優れた低保磁力希土類樹脂磁石が望まれていた。

#### 【0015】

【課題を解決するための手段】本発明は、化学量論組成 (Nd<sub>2</sub>Fe<sub>14</sub>B<sub>x</sub>) 付近の Nd<sub>2</sub> ± 0.5at%、保磁力 H<sub>c1</sub>, ≈10kOe の急冷粉体と、結晶粒が 20~50nm に制御されたソフト磁性相とハード磁性相からなる交換スプリング磁石粉体とを混合し、樹脂で固めて低保磁力希土類樹脂磁石とする。また磁石粉体の充填密度をできる限り高めるために 32μm 以下を含む交換スプリング磁石粉体を、前記磁石粉体よりも粉体粒子径が大きな Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成 (Nd<sub>2</sub>Fe<sub>14</sub>B<sub>x</sub>) 付近の急冷粉体と混合し、これを樹脂でリング状に固めバーミアンス係数 P<sub>C</sub> を 5 以上とした低保磁力希土類樹脂磁石とすると磁気特性や熱安定性の整合性を高めるのに効果的である。

#### 【0016】

【作用】Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成よりも低 Nd量での磁気特性の低下原因と考えられる αFe の析出は、結晶粒間の交換相互作用を弱めて磁化反転しやすくするためである。交換相互作用とは近接した磁気モーメントの向きを互いに揃えようとする作用で、向きが揃えば磁化は増大する。また交換相互作用は結晶粒表面を介して行われるから、その強さは粒子の表面積に依存する。同体積で考えると結晶粒径が小さい方が表面積が増え交換相互作用は強くなる。20~50nm に結晶粒が制御され、ソフト磁性相とハード磁性相からなる交換スプリング磁石粉体は、その結晶粒表面積が増えることで交換相互作用が強くなる。Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成 (Nd<sub>2</sub>Fe<sub>14</sub>B<sub>x</sub>) 付近の Nd<sub>2</sub> ± 0.5at%、保磁力 H<sub>c1</sub>, ≈10kOe の急冷粉体と結晶粒が 20~50nm に制御されたソフト磁性相とハード磁性相から構成される交換スプリング磁石粉体とを混合し、これを樹脂で固めて希土類樹脂磁石とすると低保磁力領域であっても (BH) max, Br, ΔBr/ΔT (%/K), ΔH<sub>c1</sub>/ΔT (%/K) など磁気特性の整合性の高い希土類樹脂磁石を容易に得ることができる。

#### 【0017】

【実施例】以下、本発明を実施例により説明する。

【0018】図1は Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成 (Nd量 12 at%) に対して Dyを含めた希土類元素量を 4.5at%とした低 Nd量合金組成 Nd<sub>2.5</sub>Dy<sub>0.5</sub>Fe<sub>14</sub>B<sub>11.5</sub> OPTIMUM-QUENCH 急冷粉体の Cu-Kα の X線回折图形を示す。Nd<sub>2</sub>Fe<sub>14</sub>B とともに 2θ = 43.03deg に Fe<sub>2</sub>B (220) と 2θ = 44.67deg に αFe (110) の強い回折が認められる。

急冷粉体は 20~50nm の結晶粒から構成される微細組織で約 30% の Nd<sub>2</sub>Fe<sub>14</sub>B 相、65% 以上の Fe<sub>2</sub>B 相、5% 以下の αFe 相になっている。急冷粉体の磁気特性は H<sub>c1</sub>, ≈320kA/m (~4kOe)、Br ≈ 1.2T、(BH) max ≈ 95kJ/m<sup>3</sup> (12MGOe) である。

【0019】図2は上記低 Nd量急冷粉体と Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成の Nd<sub>2</sub>Fe<sub>14</sub>B<sub>x</sub> OPTIMUM-QUENCH 急冷粉体を樹脂で固めた希土類樹脂磁石の初磁化曲線とリコイル特性を示す。Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成付近の急冷粉体の初磁化曲線が pinning 型であるのに対し、低 Nd量急冷粉体は Nucleation 型である。

【0020】図3は上記低 Nd量急冷粉体と Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成の Nd<sub>2</sub>Fe<sub>14</sub>B<sub>x</sub> OPTIMUM-QUENCH 急冷粉体の不可逆磁化反転率の印加磁界 H<sub>m</sub> 依存性を示す。ここで不可逆磁化とは逆磁界をかけたとき磁化反転しない磁化 J<sub>irrev</sub> である。J<sub>irrev</sub> は最大で残留磁化 J<sub>r</sub> の 2 倍以内であるから、J<sub>irrev</sub>/2J<sub>r</sub> を不可逆磁化反転率とした。Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成付近の急冷粉体は H<sub>c1</sub> 程度の磁界を加えると既に不可逆的に磁化反転を起こしている。一方、低 Nd量急冷粉体は H<sub>c1</sub> 程度の磁界を加えてもまだ可逆的に磁化反転している。可逆的に磁化反転するということは H<sub>c1</sub> 程度までの範囲内ならば逆磁界をなくしたときに最初の磁化の状態に戻ることである。この特徴は Kneller らのいうところの “Exchange-spring-magnet” すなわち交換スプリング磁石粉体である。

【0021】図4は粉体粒子径を異にする交換スプリング磁石粉体、および Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成の Nd<sub>2</sub>Fe<sub>14</sub>B<sub>x</sub> OPTIMUM-QUENCH 急冷粉体の 2 種を樹脂

で固めた希土類樹脂磁石の減磁曲線を示す。

【0022】Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成付近の急冷粉体は Nd<sub>2</sub>Fe<sub>14</sub>B 結晶粒径 100~300nm であるため粉体粒子径が 32μm 以下で Br, H<sub>c1</sub> および角型性 (Hk/H<sub>c1</sub>) が大きく低下する。しかし、交換スプリング磁石粉体は結晶粒径が 20~50nm と 1/2 以下であるため 32μm 以下の微粉であっても安定した磁気特性を維持する。32μm 以下を含む交換スプリング磁石粉体を、前記磁石粉体よりも粉体粒子径が大きな Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成 (Nd<sub>2</sub>Fe<sub>14</sub>B<sub>x</sub>) 付近の急冷粉体と混合することで、磁石粉体の充填密度を高めた希土類樹脂磁石を得ることができ

る。

【0023】図5は交換スプリング磁石粉体で Nd<sub>2</sub>Fe<sub>14</sub>B 化学量論組成の Nd<sub>2</sub>Fe<sub>14</sub>B<sub>x</sub> OPTIMUM-QUENCH 急冷粉体の一部を置換した混合急冷粉体を樹脂で固めた希土類樹脂磁石の磁気特性を、交換スプリング磁石粉体の置換量に対して示す。

【0024】図5のように、広範囲で希土類樹脂磁石の H<sub>c1</sub> を調整することができる。また Fe<sub>2</sub>B と αFe のキュリー温度 T<sub>C</sub> は、それぞれ 783K, 1043K で Nd<sub>2</sub>Fe<sub>14</sub>B の 585K より高いので交換スプリング磁石粉体の温度係数

5

△Br/△Tは-0.07%/K程度である。この値は、Nd<sub>2</sub>F<sub>e</sub><sub>1</sub>B化学量論組成付近の急冷粉体においてFeを16at%程度のCoで置換した場合と同水準の特性である。

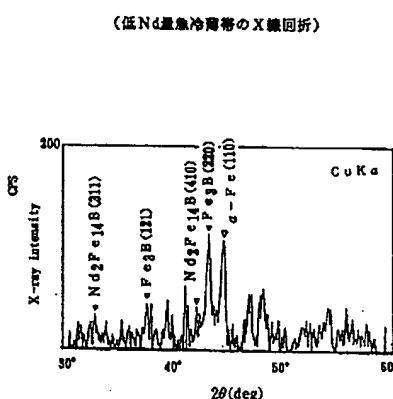
【0025】図6は上記混合粉体を樹脂で固めた希土類樹脂磁石と、合金組成  $Nd_{0.8}Fe_{0.2}$  bal  $B_0$  OPTIMUM-QUENCH急冷粉体を樹脂で固めた希土類樹脂磁石の  $H_c$ に対する  $B_r$ と  $(BH)_\text{max}$ の関係を示す。 $Nd_0Fe_{0.2}B_0$  化学量論組成付近の  $H_c$ 、795kA/m(10kOe)程度で  $(BH)_\text{max}$ は最高値を得る。しかし、それより低  $H_c$ 領域での  $B_r$ をみると、交換スプリング磁石粉体で置換した希土類樹脂磁石の方が高い  $B_r$ を示す傾向にある。

【0026】図7は交換スプリング磁石粉体、およびNd<sub>2</sub>Fe<sub>14</sub>B化学量論組成のNd<sub>2</sub>Fe<sub>14</sub>, Co<sub>5.5</sub>, B<sub>6</sub>OPTIMUM-QUENCH急冷粉体2種を樹脂で固めた希土類樹脂磁石の磁束密度Bdと80°C1000H後の不可逆減磁率を磁石のバーミアンス係数Pcに対して示す。交換スプリング磁石粉体を樹脂で固めた希土類樹脂磁石はHc<sub>s</sub>は低いけれども高Brであるため、Pc=5以上ではNd<sub>2</sub>Fe<sub>14</sub>B化学量論組成付近の急冷粉体を樹脂で固めた希土類樹脂磁石以上の磁束が得られる。また、一般にHc<sub>s</sub>とHc<sub>r</sub>の温度係数は磁石の不可逆減磁に代表される熱安定性に影響をおよぼすが、交換スプリング磁石粉体のHc<sub>r</sub>の温度係数△Hc<sub>r</sub>/△Tは-0.36~-0.40%/Kで、この値は化学量論組成付近の急冷粉体のそれにはほぼ等しい。すなわち低H<sub>r</sub>の割合不可逆減磁率が小さい特徴がある。

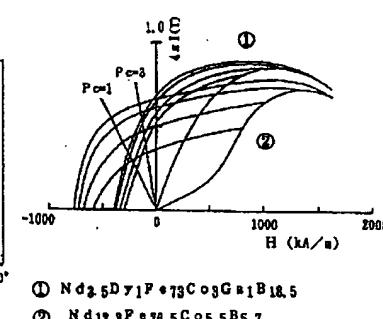
(0027)

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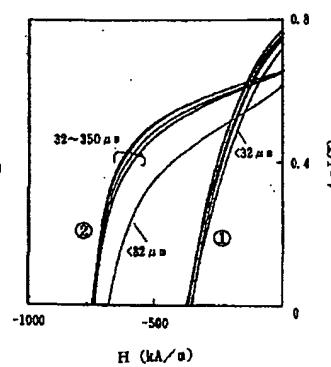
[图11]



### (低Nd量と化学量論Nd量急冷磁石の減磁曲線の印加磁場依存性)



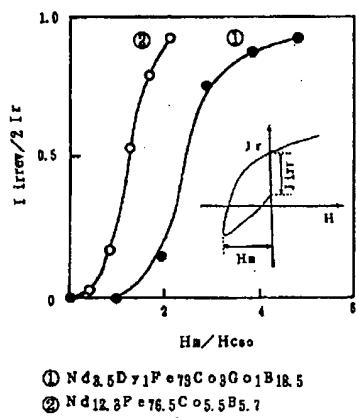
### (低Nd量と化学量論Nd量急冷磁石の 複素粒子径による極偏曲率の変化)



① Nd<sub>2.5</sub>Dy<sub>1</sub>F<sub>6</sub>γ<sub>3</sub>C<sub>0.3</sub>O<sub>1.1</sub>B<sub>18.5</sub>  
② Nd<sub>12.3</sub>F<sub>6</sub>γ<sub>8.5</sub>C<sub>0.5</sub>5.5B<sub>5.7</sub>

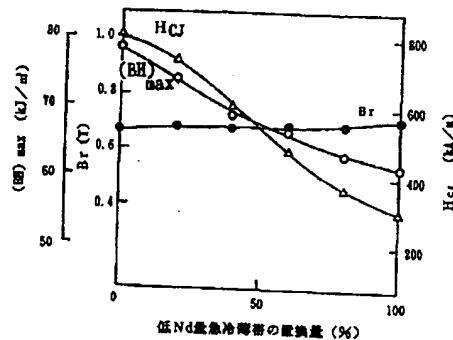
【図3】

(低Nd量と化学量論Nd量急冷磁石の不可逆磁化反転率の印加磁界依存性)



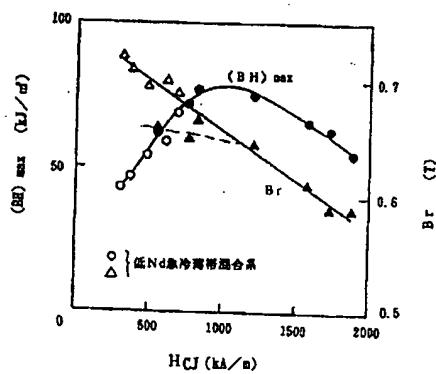
【図5】

(低Nd量と化学量論Nd量との混合急冷磁石の磁気特性)



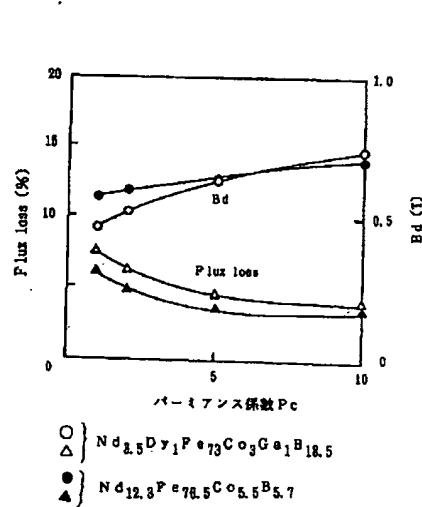
【図6】

(急冷磁石のHcとBH<sub>max</sub>, Brとの関係)



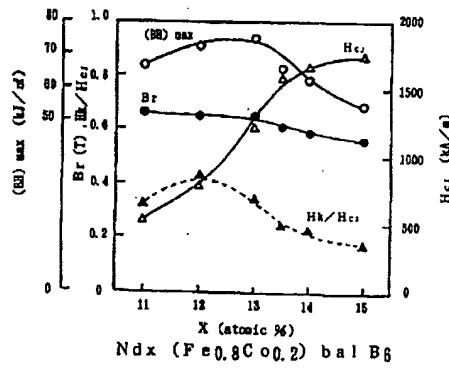
【図7】

(低Nd量と化学量論Nd量急冷磁石の磁束量と不可逆減磁のパーミアンス係数依存性)

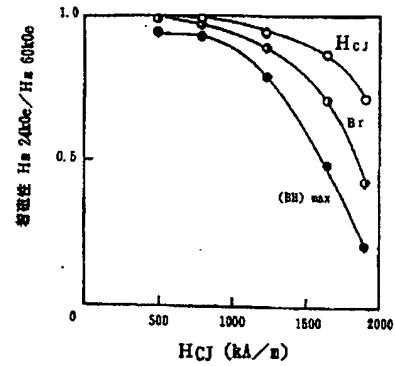


【図8】

(急冷帶のNd量と磁石特性)



【図9】

(急冷帶のH<sub>CJ</sub>と磁石特性)

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CLAIMS

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[Claim(s)]

[Claim 1] It is the low coercive force rare earth resin magnet of under 795 kA/m (10kOe) more than 320 kA/m (4kOe) that mixed the quenching fine particles of coercive force HCJ\*\*10kOe, and the exchange spring magnet fine particles which consist of a software magnetism phase by which crystal grain was controlled by 20-50nm, and a hard magnetism phase Nd12\*\*0.5at% near Nd2Fe14B stoichiometric composition (Nd12Fe82 B6), and hardened this by resin.

[Claim 2] It is the low coercive force rare earth resin magnet of under 795 kA/m (10kOe) more than 320kA (4kOe)/m that mixed the conversion spring magnet fine particles which consist of a software magnetism phase in which crystal grain contains 32 micrometers or less, and a hard magnetism phase with the quenching fine particles near [ than said exchange spring magnet fine particles / where fine-particles particle diameter is bigger ] Nd2Fe14B stoichiometric composition (Nd12Fe82 B6), and hardened this in the shape of a ring by resin.

[Claim 3] The low coercive force rare earth resin magnet according to claim 1 or 2 which made the permeance coefficient Pc five or more.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the low coercive force rare earth resin magnet which mixes the exchange spring magnet fine particles which consist of quenching fine particles of coercive force HCJ\*\*10k0e, a software magnetism phase by which crystal grain was controlled by 20-50nm, and a hard magnetism phase, and is hardened by resin Nd12\*\*0.5at% near Nd2Fe14B stoichiometric composition (Nd12Fe82B6).

[0002]

[Description of the Prior Art] The quenching fine particles which carried out the melt span of Nd of the rate near 2:14:1, Fe, and the B ternary system molten metal alloy are the ingredients of the metastable state which makes the main phase the Nd2Fe14B compound of about 870 crystallization temperature K, are classified into OVER-QUENCH, OPTIMUM-QUENCH, and a UNDER-QUENCH condition according to extent of quenching, and serve as high coercive force (HCJ) in the state of OPTIMUM-QUENCH.

[0003] J. F. As Rare earth-Iron-Boron Materials:A New Era in PermanentMagnets, Ann. Rev. Mater. Sci., and vol. 16 p467 (1986) have described Herbest and others, the residual magnetization Jr of the quenching thin band of the OPTIMUM-QUENCH condition which carried out crystal control makes the Nd2Fe14 B phase 0.8T and maximum energy product ([BH] max) 111.5 kJ/m<sup>3</sup> (14MGOe) less than [ single domain critical size -300nm ].

[0004] Generally it compresses into a ring configuration to about 80% of true density chiefly, fixes by resin, and let the quenching fine particles which ground the quenching thin band of the above and an OPTIMUM-QUENCH condition in mean particle diameter of about 150 micrometers be the rare earth resin magnet of (BH) max - 71 kJ/m<sup>3</sup> (- 9MGOe). This rare earth resin magnet was put in practical use quickly [ since / 1987 ], and acquired the status as a rare earth resin magnet following a SmCo system.

[0005] On the other hand, research of the quenching thin band of the amount of low Nd(s) was also done rather than Nd2Fe14B stoichiometric composition (Nd12Fe82 B6) with progress of utilization of quenching fine particles.

[0006] a system [ in / Hirosawa and others is High Coercivity Iron-Rich Rare earth Permanent Magnet Material Based on(Fe, Co) 3 B-Nd-

M(M=aluminum, Si, Cu, Ga, Ag, Au). 37th MMM(1992) FC-10, and / from Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition / the amount of low Nd(s) ] -- Ga etc. -- 1at% -- the quenching fine particles of an OPTIMUM-QUENCH condition, then HCJ suppose that 320 kA/m (4kOe) extent will be obtained by adding and carrying out detailed control of the crystal grain.

[0007] The point that the ratio of residual magnetization Jr and saturation magnetization Js is not about 0.50, for example, is high with Jr/Js=0.75 with isotropy magnetically attracts attention to the amount quenching fine particles of low Nd(s) of Nd \*\*\*\* 4at%.

[0008] It was presupposed that HCJ came out the main phase of the above-mentioned quenching fine particles by being connected in the exchange interaction which is a fine crystal grain (30nm) and the soft Fe<sub>3</sub>B metastable phase minded Nd<sub>2</sub>Fe<sub>14</sub> B phase and the grain boundary magnetically, and Kneller and others named the "Exchange-spring-magnet" exchange spring magnet to this, as it was in The Exchange-Spring Magnet-A New Material Principle for Permanent Magnets, IEEE Trans. Magn., and 27p3588 (1991). Software phase Fe<sub>3</sub>B and hard phase Nd<sub>2</sub>Fe<sub>14</sub>B are connected in an exchange interaction, and unless a hard phase carries out flux reversal, even if the origin of a name removes an opposing magnetic field, it is in the point that magnetization of a software phase returns reversibly like a spring (spring), and the original magnetization is obtained. If a hard phase and a software phase usually exist in this way, a stage will be generated in a magnetization curve and the fall of magnetic properties will be caused. However, if crystal grain is made detailed so that an exchange interaction works to two interphases (20-50nm), in spite of being isotropy, it is clear that high Jr is obtained, without producing a stage.

[0009]

[Problem(s) to be Solved by the Invention] Drawing 8 shows the magnetic properties over the amount of Nd(s) of the rare earth resin magnet which hardened alloy presentation Ndx(Fe0.8Co0.2) bal B6OPTIMUM-QUENCH quenching fine particles by resin. It is \*\* and is a magnetic-properties value after field (Hm) 4770 kA/m pulse magnetization. Moreover, Hk is a demagnetizing field when magnetization reaches 90% of Br, and Hk/HCJ expresses the square shape nature of a demagnetization curve.

[0010] Although the amount of Nd(s) in Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition is 12at(s)% , magnetic properties, such as (BH) max, Hk/HCJ, and Br, have the highest adjustment at 12 - 12.5at% near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition. Although it will be set to high HCJ if the amount of Nd(s) exceeds 12.5at(s)% , (BH) max, Hk/HCJ, and Br fall.

[0011] Drawing 9 shows the ratio of magnetic properties, such as field

(H<sub>m</sub>) 1908 kA/m of the above-mentioned rare earth resin magnet, (BH) max after 4770 kA/m pulse magnetization, H<sub>k</sub>/HCJ, and Br, with relation with HCJ.

[0012] If HCJ exceeds 795 kA/m (10kOe), magnetic magnetization nature will fall. Therefore, generally a thing with the rare earth resin magnet near the stoichiometric composition of HCJ795 kA/m (10kOe) extent with carrying out [ much ] multi-electrode magnetization is used.

[0013] However, if the distance between electrodes narrows further, it will become difficult to generate sufficient magnetization field and magnetization will become difficult also by HCJ795 kA/m (10kOe).

Moreover, if it is made the amount of low Nd(s) like [ stoichiometric composition / Nd<sub>2</sub>Fe<sub>14</sub>B ] drawing 3 , the deposit of alphaFe will be pressed down, and since it becomes difficult to attain homogenization of an organization, magnetic properties, such as HCJ, (BH) max, H<sub>k</sub>/HCJ, and Br, fall.

[0014] as mentioned above, (BH) -- a low coercive force rare earth resin magnet excellent in multi-electrode magnetization nature was desired, without reducing magnetic properties, such as max, Br, deltaBr/deltaT (%/K), and deltaHCJ/deltaT (%/K), as much as possible.

[0015]

[Means for Solving the Problem] Nd<sub>12</sub>\*\*0.5at% near stoichiometric composition (Nd<sub>12</sub>Fe<sub>82</sub> B<sub>6</sub>), this invention mixes the quenching fine particles of coercive force HCJ\*\*10kOe, and the exchange spring magnet fine particles which consist of a software magnetism phase by which crystal grain was controlled by 20-50nm, and a hard magnetism phase, hardens them by resin, and let it be a low coercive force rare earth resin magnet. Moreover, if it is the low coercive force rare earth resin magnet which mixed the exchange spring magnet fine particles containing 32 micrometers or less with the quenching fine particles near [ than said magnet fine particles / where fine-particles particle diameter is bigger ] Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition (Nd<sub>12</sub>Fe<sub>82</sub> B<sub>6</sub>), hardened this in the shape of a ring by resin, and made the permeance coefficient P<sub>c</sub> five or more in order to raise the pack density of magnet fine particles as much as possible, it is effective for raising magnetic properties and the adjustment of thermal stability.

[0016]

[Function] The deposit of alphaFe considered to be the cause of a fall of the magnetic properties in the amount of low Nd(s) rather than Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition is for making flux reversal easy to weaken the exchange interaction between crystal grain and to carry out. An exchange interaction is an operation which is going to arrange the

sense of the magnetic moment which approached mutually, and if the sense gathers, magnetization will increase. Moreover, since an exchange interaction is performed through a crystal grain front face, the strength is dependent on the surface area of a particle. If it thinks by this volume, surface area of the one where the diameter of crystal grain is smaller will increase, and an exchange interaction will become strong. Crystal grain is controlled by 20-50nm, and an exchange interaction becomes strong because the crystal grain surface area of exchange spring magnet fine particles which consist of a soft magnetism phase and a hard magnetism phase increases. Nd<sub>12</sub>\*\*0.5at% near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition (Nd<sub>12</sub>Fe<sub>82</sub> B<sub>6</sub>), The exchange spring magnet fine particles which consist of quenching fine particles of coercive force HCJ\*\*10kOe, a soft magnetism phase by which crystal grain was controlled by 20-50nm, and a hard magnetism phase are mixed. When this is hardened by resin and it is a rare earth resin magnet, even if it is a low coercive force field -- (BH) -- a rare earth resin magnet with the high adjustment of magnetic properties, such as max, Br, deltaBr/deltaT (%/K), and deltaHCJ/deltaT (%/K), can be obtained easily.

[0017]

[Example] Hereafter, an example explains this invention.

[0018] Drawing 1 shows the X-ray diffraction pattern of Cu-Kalpha of the amount alloy presentation Nd of low Nd<sub>3.5</sub>Dy<sub>1</sub>Fe<sub>73</sub>Co<sub>3</sub>Ga<sub>1</sub>B<sub>18.5</sub> OPTIMUM-QUENCH quenching fine particles which made 4.5at(s)% the amount of rare earth elements which includes Dy to Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition (amount of Nd(s) 12at%). The strong diffraction of alphaFe (110) is accepted to be Fe<sub>3</sub>B (220) to 2theta=43.03deg by 2theta=44.67deg with Nd<sub>2</sub>Fe<sub>14</sub>B. Quenching fine particles have about 30% of Nd<sub>2</sub>Fe<sub>14</sub>B phase, 65% or more of Fe<sub>3</sub>B phase, and 5% or less of alphaFe phase in the detailed organization which consists of 20-50nm crystal grain. The magnetic properties of quenching fine particles are HCJ - 320 kA/m (- 4 kOe), Br-1.2T, and (BH) max - 95 kJ/m<sup>3</sup> (12MG0e).

[0019] Drawing 2 shows the initial magnetization curve and recoiling property of a rare earth resin magnet of having hardened the above-mentioned amount quenching fine particles of low Nd(s), and the Nd<sub>12</sub>Fe<sub>76.5</sub>Co<sub>5.50</sub>B<sub>6</sub>OPTIMUM-QUENCH quenching fine particles of Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition by resin. The amount quenching fine particles of low Nd(s) are Nucleation molds to the initial magnetization curve of the quenching fine particles near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition being a pinning mold.

[0020] Drawing 3 shows the impression field H<sub>m</sub> dependency of the rate of irreversible flux reversal of the above-mentioned amount quenching fine

particles of low Nd(s), and the Nd<sub>12</sub>Fe<sub>76.5</sub>Co<sub>5.50</sub> B<sub>6</sub>OPTIMUM-QUENCH quenching fine particles of Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition. Irreversible magnetization is the magnetization J<sub>irrev</sub> which does not carry out flux reversal here, when an opposing magnetic field is applied. Since J<sub>irrev</sub> was less than 2 double [ of residual magnetization J<sub>r</sub> ] at the maximum, it made J<sub>irrev</sub>/2J<sub>r</sub> the rate of irreversible flux reversal. The quenching fine particles near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition have already started flux reversal irreversibly, if the field of HCJ extent is added. On the other hand, even if the amount quenching fine particles of low Nd(s) add the field of HCJ extent, flux reversal of them is still carried out reversibly. Carrying out flux reversal reversibly is returning to the condition of the first magnetization, when it was within the limits to HCJ extent and an opposing magnetic field is lost. This description is, "Exchange-spring-magnet, i.e., the exchange spring magnet fine particles," which Kneller and others says. [0021] Drawing 4 shows the demagnetization curve of a rare earth resin magnet which hardened two sorts of the Nd<sub>12</sub>Fe<sub>76.5</sub>Co<sub>5.50</sub> B<sub>6</sub>OPTIMUM-QUENCH quenching fine particles of the exchange spring magnet fine particles which differ in fine-particles particle diameter, and Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition by resin.

[0022] Since the quenching fine particles near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition are 100–300nm of diameters of Nd<sub>2</sub>Fe<sub>14</sub>B crystal grain, Br, HCJ, and square shape nature (H<sub>k</sub>/HCJ) fall [ fine-particles particle diameter ] greatly by 32 micrometers or less. However, exchange spring magnet fine particles maintain the magnetic properties stabilized even if it was fines 32 micrometers or less, since the diameters of crystal grain were 20–50nm and 1/2 or less. The rare earth resin magnet which raised the pack density of magnet fine particles can be obtained by mixing the exchange spring magnet fine particles containing 32 micrometers or less with the quenching fine particles near [ than said magnet fine particles / where fine-particles particle diameter is bigger ] Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition (Nd<sub>12</sub>Fe<sub>82</sub> B<sub>6</sub>).

[0023] Drawing 5 shows the magnetic properties of the rare earth resin magnet which hardened the mixed quenching fine particles which permuted some Nd<sub>12</sub>Fe<sub>76.5</sub>Co<sub>5.50</sub> B<sub>6</sub>OPTIMUM-QUENCH quenching fine particles of Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition by resin to the amount of permutations of exchange spring magnet fine particles by exchange spring magnet fine particles.

[0024] Like drawing 5 , it is wide range and HCJ of a rare earth resin magnet can be adjusted. Moreover, since Curie-temperature T<sub>c</sub> of Fe<sub>3</sub>B and alphaFe is higher than 585K of Nd<sub>2</sub>Fe<sub>14</sub>B respectively 783K and 1043K, T

is -0.07% of temperature coefficient delta Br/delta T of exchange spring magnet fine particles, and about K. This value is a property equivalent to the case where Fe is permuted by about [ 16at% ] Co in the quenching fine particles near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition.

[0025] Drawing 6 shows the relation between Br to HCJ of the rare earth resin magnet which hardened the above-mentioned mixed fine particles by resin, and the rare earth resin magnet which hardened alloy presentation Nd<sub>x</sub>(Fe0.8Co0.2) bal B60OPTIMUM-QUENCH quenching fine particles by resin, and (BH) max. (BH) max obtains a peak price with HCJ795 kA/m (10kOe) extent near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition. However, when Br in a low HCJ field is seen from it, the direction of the rare earth resin magnet permuted by exchange spring magnet fine particles is in the inclination which shows high Br.

[0026] Drawing 7 shows the flux density Bd of the rare earth resin magnet which hardened exchange spring magnet fine particles and two sorts of Nd<sub>12</sub>Fe<sub>76.5</sub>Co<sub>5.50</sub> B60OPTIMUM-QUENCH quenching fine particles of Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition by resin, and the irreversible demagnetizing factor after 80-degree-C1000H to the magnetic permeance coefficient Pc. Although the rare earth resin magnet of HCJ which hardened exchange spring magnet fine particles by resin is low, since it is high Br, the magnetic flux more than the rare earth resin magnet which hardened the quenching fine particles near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition by resin is acquired more than by Pc=5. Moreover, generally, \*\*\*\*\* of T are temperature coefficient delta HCJ/delta T -0.36 of HCJ of exchange spring magnet fine particles - -0.40%/K about effect, and this value is almost equal to the thermal stability with which the temperature coefficient of HCJ and HCJ is represented by magnetic irreversible demagnetization to it of the quenching fine particles near stoichiometric composition. That is, there is the description that an irreversible demagnetizing factor is small considering low HCJ.

[0027]

[Effect of the Invention] Nd<sub>12\*\*0.5</sub>at% near Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition (Nd<sub>12</sub>Fe<sub>82</sub> B<sub>6</sub>), The exchange spring magnet fine particles which consist of quenching fine particles of coercive force HCJ\*\*10kOe, a soft magnetism phase by which crystal grain was controlled by 20-50nm, and a hard magnetism phase are mixed. when this is hardened by resin and it is a rare earth resin magnet, even if it is a low coercive force field -- (BH) -- the rare earth resin magnet excellent in multi-electrode magnetization nature with the high adjustment of magnetic properties, such as max, Br, deltaBr/deltaT (%/K), and deltaHCJ/deltaT (%/K), can be obtained easily. Moreover, if the exchange spring magnet

fine particles containing 32 micrometers or less are mixed with the quenching fine particles near [ than said magnet fine particles / where fine-particles particle diameter is bigger ] Nd<sub>2</sub>Fe<sub>14</sub>B stoichiometric composition, the rare earth resin magnet which raised the pack density of magnet fine particles can also be obtained.

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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] The property Fig. showing an X-ray diffraction pattern

[Drawing 2] The initial magnetization curve and recoiling property Fig. of a rare earth resin magnet

[Drawing 3] The property Fig. showing the impression field dependency of the rate of irreversible flux reversal

[Drawing 4] The property Fig. showing the demagnetization curve of a rare earth resin magnet

[Drawing 5] The magnetic-properties Fig. to the amount of permutations of exchange spring magnet fine particles

[Drawing 6] The property Fig. showing the relation between HCJ and Br(BH) max

[Drawing 7] A permeance coefficient, flux density, the property Fig. showing the relation of an irreversible demagnetizing factor

[Drawing 8] The magnetic-properties Fig. to the amount of Nd(s) of a rare earth resin magnet

[Drawing 9] The property Fig. showing HCJ and magnetization nature of a rare earth resin magnet

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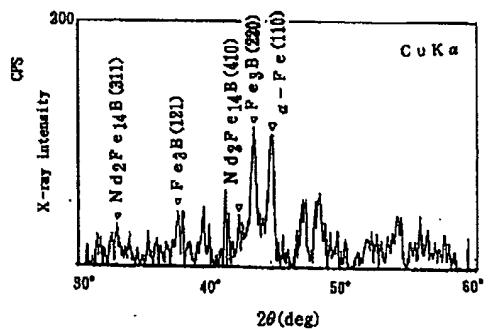
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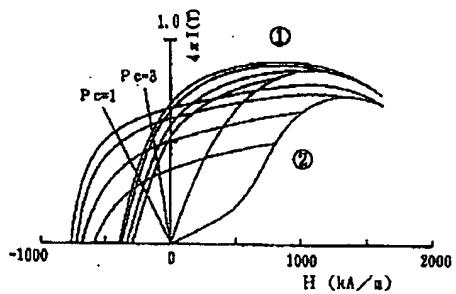
DRAWINGS

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[Drawing 1]  
(低Nd量急冷帶のX線回折)



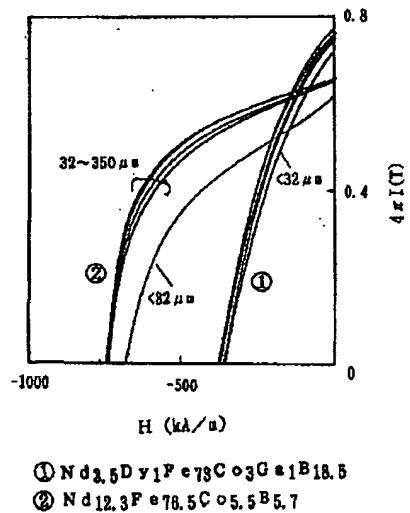
[Drawing 2]  
(低Nd量と化学量論Nd量急冷磁石の減磁曲線の印加電界依存性)



① Nd<sub>3.5</sub>Dy<sub>1</sub>Fe<sub>13</sub>C<sub>0.3</sub>Ga<sub>1</sub>B<sub>18.5</sub>  
② Nd<sub>12.8</sub>Fe<sub>76.5</sub>C<sub>0.5</sub>B<sub>5.7</sub>

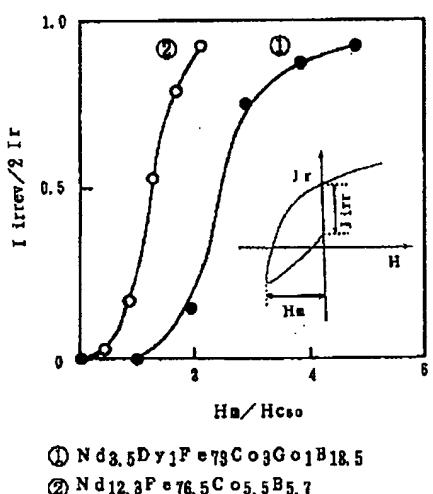
[Drawing 4]

(低Nd量と化学量論Nd量急冷磁石の  
南帯粒子径による減磁曲線の変化)



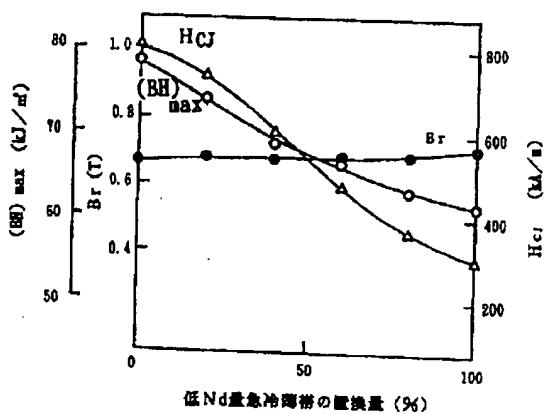
[Drawing 3]

(低Nd量と化学量論Nd量急冷磁石の  
不可逆磁化反転率の印加磁界依存性)

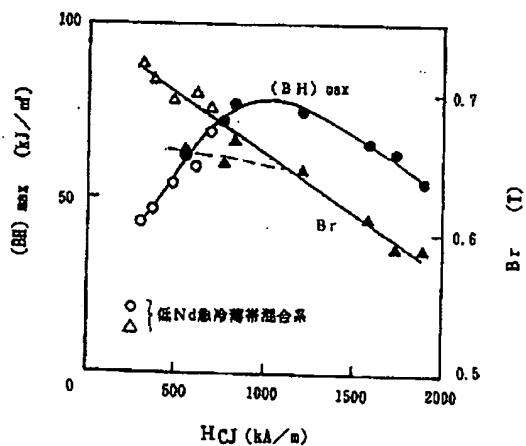


[Drawing 5]

(低Nd量と化学量論Nd量との  
混合急冷磁石の磁気特性)

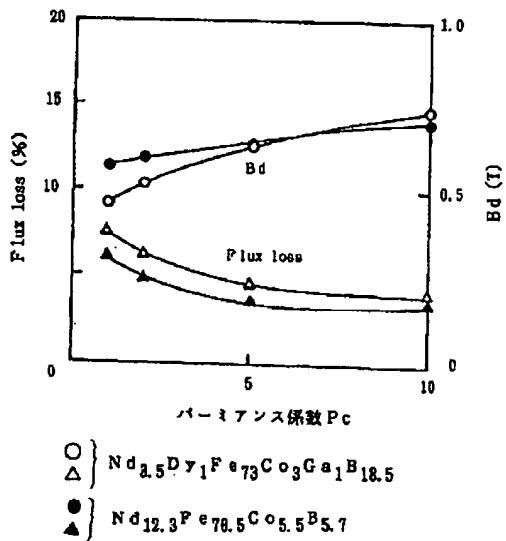


[Drawing 6]  
(急冷磁石の  $H_{cJ}$  と  $(BH)_{max}$ ,  $B_r$  との関係)

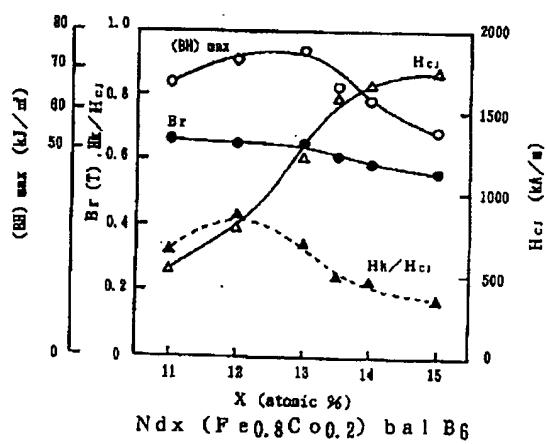


[Drawing 7]

(低Nd量と化学量論Nd量急冷磁石の磁束量と  
不可逆減磁のパーミアンス係数依存性)

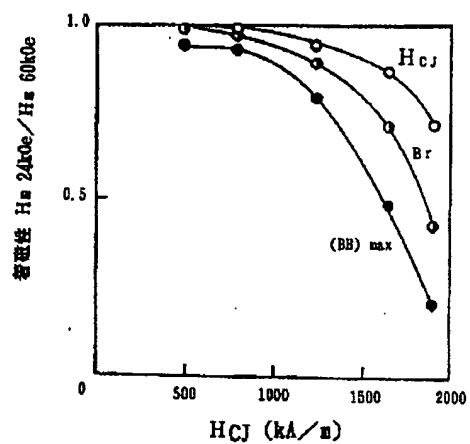


[Drawing 8]  
(急冷帶のNd量と磁石特性)



[Drawing 9]

(急冷薄帯の $H_{cJ}$ と着磁特性)



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[Translation done.]